EPA VALIDATION OF CIBA RESULTS DIDNINS

28 may 2 Ac 9109022-0A

DATA VALIDATION COVER SHEET

. Site Name:	CIBAGEI	<i>6 y</i>
. Case/SAS#:		3. SDG#: 6684A-1
. Data Valida (if appli . Data Review	tion Subcontractor cable) er:	J RARTELS.
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SEMS DocID 651251



Environmental Services Assistance Teams Region 1

19 Crosby Drive Bedford, Massachusetts 01730

Ms. Margaret Leshen
Regional Sample Control Custodian
U.S. Environmental Protection Agency
90 Canal Street
Boston, Massachusetts 02114

February 6, 1992 Revised 4/8/92 B-92-04-Y

Re: TID No. 01-92-01-15

SAS No. 6684A, SDG No. 6684A-1

Reystone / NEA Environmental Resources
CIBA GEIGY Cranston, RI.

BO# 9109022-0A

Dioxin: /4/Aqueous/6684A-3,6684A-4,6684A-5,6684A-6 PEM/2/Soil/6684A-1,6684A-2

Dear Ms. Leshen:

A validation was performed on the dioxin/furan analytical data for 4 aqueous samples and 2 PEM samples collected at the Ciba-Geigy site in Cranston, RI. The samples were analyzed according to SAS 6684A Specifications. The data were evaluated based on the following parameters:

- Data Completeness
- * PEM Samples
 - Window Defining Mix
- calibration
- * Column Performance
- * Internal Standards
- * Recovery Standards
 - Method Blanks
 - Duplicate Samples
 - Matrix Spike/Matrix Spike Duplicates
- Concentration/EMPC/EDL
- * Total Congener Concentrations
 - Toxic Equivalent Factors
- * All criteria were met for this parameter

Table 1 summarizes the validation recommendations which were based on the following information:

Please contact Janine Bartels at the Lockheed ESAT office at (617) 275-7868 should you have any questions or comments regarding the information.

Very truly yours,

LOCKHEED ENGINEERING & SCIENCES COMPANY

Janine Bartels Senior Scientist

Jack Berges ESAT Team Manager

/jba Enclosures cc: Steve Stodola

ATTACHMENT I



DIOXIN DATA REVIEW WORKSHEET

Regional Review	of	Dioxin	Data	Pac	kage
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Regional Review of Dioxin Data Package					
The hard copied (laboratory name <u>Reystone</u> NEA data package received at Region I has been reviewed and the quality assurance and performance data summarized. The data review included:					
Case No. (684A) Sampling Date 9 11 /91 Matrix Water Date Received by Laboratory	SAS No. 66844 No. of Samples 12 Shipping Date 9/11/9				
Sample Nos.:					
PEM Nos. Laboratory Fortified Std 9/45/25 Fortified Blank 9/45/25	ID EPA Identification No. <u>SP02-02</u> <u>WEE 656 6684</u> A-2 <u>P62-01</u> 083 D4 <u>T</u> 6684 A				
The general criteria use based on an examination of:	d to determine the performance were				
o PEM Samples o Initial and continuing calibrations o Retention time marker solutions o Estimated maximum possible concentration	o Method blanks o Instrument sensitivity check o Chromatographic resolution				
Definition of Qualifiers A - Acceptable data J - Approximate data due to quality control criteria R - Reject data due to quality control criteria U - Not detected S - [M-COCl] + ion did not meet S/N ratio >2.5 requirement H - Did not meet the ion abundance criteria					

Ms. Leshen Page 2

Data Completeness

The laboratory was contacted on January 23, 1992 and on January 28, 1992 for the following information: sample calculations of the estimated detection limits (EDL); sample calculations of the estimated maximum possible concentration (EMPC); and sample calculations of the concentration for sample 6684A-2; confirmation of TEF values for 2,3,4,7,8-PeCDF and the adjusted concentrations; clarify the identity of EPA 6684A-6 (tag#12913 and tag#12914) which did not match Keystone ID# listed in the SDG narrative; corrected PCDD forms for sample 6684A-6; form 1 PCDD-3 for sample 6684A-5/12910; autospec 1 instrument log notes for 10/19/91, pages 31 and 32; and an explanation of what happened to the original pink chain of custody forms. Information was received from the laboratory on February 4, 1992. Phone logs and correspondence are enclosed.

PEM Samples

No 2,3,7,8-TCDD was reported in the blank PEM sample 6684A-1 (083D4I). In the spike PEM sample 6684A-2 (WEE656), 1.8 ppb of 2,3,7,8-TCDD was reported which is within the 99% prediction limit.

Window Defining Mix

The analyte 2,3,7,8-TCDF shifted outside of the required retention time windows for sample 6684A-3. Since the analyte did not meet rentention time criteria for the 2,3,7,8-TCDF analyte, estimate (J) positive values for 2,3,7,8-TCDF for sample 6684A-3.

Internal and Recovery Standards

The laboratory spiked the sample extract with 200 pg of 37C-2,3,7,8-TCDD immediately prior to the clean-up procedure, rather than 800 pg as required in EPA method 1613. This was to reduce contamination in the m/z=322 channel. No action was required. All other criteria for internal and recovery standards were met.

Blanks

DFBLK1 was lost during the evaporation process because the flask cracked. Estimate (J) positive results for sample 6684A-3 associated with this blank.

DFBLK2 showed low level of OCDD/OCDF. Estimate (J) positive for OCDD/OCDF for all samples due to low level OCDD/OCDF contamination throughout this SDG.

DFBLK4 was contaminated by the laboratory during the sample preparation process. Estimate (J) positive results for all analytes for sample 6684A-6 associated with this blank.

Ms. Leshen Page 3

<u>Duplicate Samples</u>

Samples 6684A-3 and 6684A-3D showed %RPD values outside the 50-150% range for 1,2,3,6,7,8-HxCDF, 1,2,3,4,7,8-HxCDD, 1,2,3,6,7,8-HxCDD and 1,2,3,7,8,9-HxCDD. Estimate (J) positive results and (UJ) non-detects. Samples 6684A-5 and 6684A-5D showed %RPD values outside the 50-150% range for 2,3,4,6,7,8-HxCDF and 1,2,3,4,7,8,9-HpCDF. Estimate (J) positive results and (UJ) non-detects.

Sample 6684A-6: The data in the original sample packet was labeled #91US12SP02-11. The sample was actually the duplicate of that sample #91US12SP02-12. The first sample #91US12SP02-11 was contaminated during extraction. No duplicate sample was run. No action was necessary.

Matrix Spike and Matrix Spike Duplicate

Recoveries for 2,3,4,7,8-PeCDF (165%) and OCDF (46%) were outside the 60-140% limits in sample 6684A-3S. Estimate (J) positive values and (UJ) non-detects for OCDF and 2,3,4,7,8-PeCDF for the unspiked 6684A-3 samples.

Concentration / EMPC / EDL

Estimated (J) values will be reported as EMPC for parameters which have blank contamination, retention time window shifts, % RPD outside limits and % recoveries outside limits.

Toxic Equivalent Factors

Toxic Equivalent Factors used to calculate TEF adjusted concentrations are from "Interim Procedures for Estimating Risk Associated with Exposure to Mixtures of Chlorinated Dibenzo-p-dioxins and Dibenzofurans (CDDs and CDFs)" EPA 625-3-89-016, March 1989. The laboratory used a multiplying factor of 0.05 for 2,3,4,7,8-PeCDF. The multiplying factor should have been 0.5. The laboratory resubmitted corrected TEFs as requested.

Data Package Summary

The overall quality of the dioxin data package was acceptable. The DC-2 forms and page numbering were not included in the original package. The laboratory submitted DC-2 forms with no page numbers. No other problems were encountered with this case.

Data Summary Key

- A Acceptable data.
- J The associated numerical value is an estimated quantity.
- R Reject data due to quality control criteria. The data are unusable (compound may or may not be present). Resampling and reanalysis is necessary for verification.
- U The compound was analyzed for, but was not detected. The associated numerical value is the sample quantitation limit.
- UJ The compound was analyzed for, but was not detected. The sample quantitation limit is an estimated quantity.
- -- The compound was analyzed for, but was not detected. The sample quantitation limit is the same as the CRQL presented.

SITE: Ciba - Geigy - Cranston, RI

CASE/SAS NO: 6684A / 6684A-1

Sample No	6684A-1/1	2902	66844-2/1	2901	6684A-3/1	2903
Natrix	Soi		Soil	l.	Vate	er
TCDD/TCDF Conc	pg/g	DL/EMPC*	pg/9	DL/EMPC*	pg/L	DL/EMPC*
2,3,7,8-TCDD	U	0.45	1800		UJ .	5.2
1,2,3,7,8-PeCDD	U	0.72	U	0.65	9.0 J	
1,2,3,4,7,8-HxCDD	U	3.1	U	12	UJ	1.2
1,2,3,6,7,8-HxCDD	Ü	2.1	Ų	8.0	กา	9.0
1,2,3,7,8,9-HxCDD	U	1.3	. <u>U</u>	3.9	n?	3.0
1,2,3,4,6,7,8-HpCDD	100		100		56 J	<u> </u>
OCDD	140 J		3600 J		630 J	
2,3,7,8-TCDF	U	0.41	U	0.43	240 J	<u> </u>
1,2,3,7,8-PeCDF	U	1.9	υ	0.80	4.8 J	
2,3,4,7,8-PeCDF	U	1.1	U	0.72	46 J	1
1,2,3,4,7,8-HxCDF	U	1.1	U	5.2	24 J	
1,2,3,6,7,8-HxCDF	U	1.1	U	5.1	7.1 J	
2,3,4,6,7,8-HxCDF	U	2.5	U		7.3 J	
1,2,3,7,8,9-HxCDF	U	1.2	U	7.0	บง	1.6
1,2,3,4,6,7,8-HpCDF	Ų	950	U	2700	บู	32
1,2,3,4,7,8,9-HpCDF	υ	27	U	5.9	บู	7.0
OCDF	58 J		150 J	<u> </u>	39 J	
			<u> </u>		ļ	<u> </u>
TOTAL TCDD	. U	0.45	3600		120 J	
TOTAL PeCDD	U	0.72	U	0.65	9.0 J	<u> </u>
- TOTAL HXCDD	U	1.3	U	3.9	7.7 J	
TOTAL HpCDD	190		150		110 J	
TOTAL TCDF	UU	0.41	1.8		47000 J	
TOTAL PECDF	U	1.1	<u> </u>	0.72	200 J	
TOTAL HXCDF	· U	1.1	U	5.2	79 J	
TOTAL HpCDF	2700		2400		19 J	
TOXICITY EQUIVALENCY	1.2	pg/g J	1800) pg/g J	53	pg/L J
DILUTION FACTOR		1.0		1.0		1.0
DATE OF RECEIPT	9,	/12/91	9	/12/91	9/	12/91
SAMPLE EXTRACTION DATE	10	/10/91	10)/10/91 	10)/5/91
ANALYSIS DATE	10	/18/91	10)/19/91	10	/14/91
GC/MS 1.D.UG-TRIO-1	18001	91LCB6051	18001	791LCB6061	14001	91LCB5051

SIJE: Ciba Geigy - Cranston, RI

CASE/SAS NO: 6684A / 6684A-

Sample No	6684A-30/1	12904	6684A-4/	12907	6684A-4D/	12908
Netrix	Vat	er	Vater		Vat	er
TCDD/TCDF Conc	pg/L	DL/EMPC*	pg/L	DL/EMPC*	pg/L	DL/EMPC*
2,3,7,8-TCDD	UJ	4.3	U	2.1	U	2.8
1,2,3,7,8-PeCDD	6.9 J		U	2.7	U	2.7
1,2,3,4,7,8-HxCDD	ΟJ	2.7	U	3.1	υ	3.0
1,2,3,6,7,8-HxCDD	บม	5.3	U	2.3	U	2.4
1,2,3,7,8,9-HxCDD	บม	4.2	U	2.6	U	2.7
1,2,3,4,6,7,8-HpCDD	45 J		U	3.8	3.5	
OCDD	470 J		84 J	<u> </u>	81 J	
·						
2,3,7,8-TCDF	210 J		U	1.4	U	2.7
1,2,3,7,8-PeCDF	4.7 J		U	2.1	U	2.5
2,3,4,7,8-PeCDF	38 J		U	1.7	U	2.2
1,2,3,4,7,8-HxCDF	บา	20	U	1.4	U	1.7
1,2,3,6,7,8-HXCDF	บา	15	U	1.3	U	1.6
2,3,4,6,7,8-HxCDF	บู	5.6	U	3.5	υ	4.9
1,2,3,7,8,9-HxCDF	บJ	1.1	U	3.9	U	2.2
1,2,3,4,6,7,8-HpCDF	UJ	38	U	7.9	U	13
1,2,3,4,7,8,9-HpCDF	บม	5.1	U	1.9	U	1.4
QCDF	22 J		U	3.0	10 J	
				<u> </u>		
TOTAL TCDD	120 J		U	2.1	<u> </u>	2.8
TOTAL PeCDD	26 J	<u> </u>	U	2.7	U	2.7
TOTAL HXCDD	15 J		U	2.3	U	2.4
TOTAL HpCDD	89 J	9	UU	1.4	6.0	
TOTAL TEDF	43000 J		U	1.4	27	
TOTAL PECDF	180 J		U	1.7	U U	2.2
TOTAL HXCDF	27 J	1	U	1.3	U	1.6
TOTAL HpCDF	14 J		U	1.9	U	1.4
TOXICITY	49	pg/L J	0.08	4 pg/L J	0.13	pg/L J
DILUTION FACTOR		1.0	,	1.0		1.0
DATE OF RECEIPT	9/	12/91	9/	/12/91	9,	12/91
SAMPLE EXTRACTION DATE	10)/5/91	10	0/7/91	10)/7/91
ANALYSIS DATE	10	/14/91	10	/15/91	10	/15/91
GC/MS I.D.UG-TRIO-1	140CT	91LCB5061	14001	91LCB5131	14001	91LCB5141

SITE: Ciba Geigy - Cranston, RI

CASE/SAS NO: 6684A / 6684A-1

Sample No	6684A-5/1	2910	6684A-5D/1	2912	66844-6/1	2914
	Wate		Vat	Water		er
Matrix TCDD/TCDF Conc	pg/L	DL/EMPC*	pg/L	DL/EMPC*	pg/L	DL/EMPC*
2,3,7,8-1000	U	3.2	Ü	2.1	บู	1.7
1,2,3,7,8-PeCDD	U	3.6	Ų	2.1	UJ	2.6
1,2,3,4,7,8-HxCDD	U	4.5	U	2.6	เม	10
1,2,3,6,7,8-HxCDD	U	3.6	U	2.0	กา	9.2
1,2,3,7,8,9-HxCDD	U	4.1	U	2.2	N1	9.5
1,2,3,4,6,7,8-HpCDD	- 26		21		42 J	
OCDD	350 J		320 J		200 J	
						ļ
2,3,7,8-TCDF	U	3.4	U	1.5	23 J	
1,2,3,7,8-PeCDF	U	2.5	U	2.1	21 J	
2,3,4,7,8-PeCDF	U	2.2	U	1.9	15 J	<u> </u>
1,2,3,4,7,8-HxCDF	U	2.4	U	1.1	66 J	
1,2,3,6,7,8-HxCDF	υ	2.3	U	1.1	21 J	<u> </u>
2,3,4,6,7,8-HxCDF	υ	3.0	บง	4.1	เก	45
1,2,3,7,8,9-HxCDF	U	3.3	U	1.4	เกา	3.9
1,2,3,4,6,7,8-HpCDF	U	11	U	12	100 J	
1,2,3,4,7,8,9-HpCDF	UJ	2.7	บง	2.2	28 J	
OCDF	UJ	18	17 J		190 J	
		<u> </u>			<u>. </u>	
TOTAL TCDD		3.2		2.1	UJ	1.7
TOTAL PeCDD		3.6	<u> </u>	2.1	14 J	_
TOTAL HXCDD		4.1		2.0	22 J	
TOTAL HPCDD	46	<u> </u>	17	 	74 J	
TOTAL TCDF	6.5			1.5	. 94 J	
TOTAL PeCDF		2.2	_	1.9	130 J	
TOTAL HXCDF	UJ	2.3	กา	1.1	150 J	
TOTAL HpCDF		2.7	7.6		200 J	-
					 	
TOXICITY EQUIVALENCY	0.6	1. pg/L J	0.5	5 pg/L J	- 22	pg/L J
DILUTION FACTOR		1.0		1.0		1.0
DATE OF RECEIPT	9	/12/91	9	/12/91		/12/91
SAMPLE EXTRACTION DATE	1	0/7/91	1	0/7/91	10	0/22/91
ANALYSIS DATE	10	0/15/91	10	0/15/91		0/24/91
GC/MS I.D.UG-TRIO-1	1400	T91LCB5111	1400	191LC85121	2400	T91LCB2021

CIBA GEIGY CASE/SAS: 6684A-1 TABLE I RECOMMENDATIONS SUMMARY

SAMPLE NUMBER:	6684A-1/12902	6684A-2/12901	6684A-3/12903	6684A-3D/12904	
DIOXINS			-		
2,3,7,8-TCDD 1,2,3,7,8-PeCDD 1,2,3,4,7,8-HxCDD 1,2,3,6,7,8-HxCDD 1,2,3,7,8,9-HxCDD 1,2,3,4,6,7,8-HpCDD OCDD	A A A A A J ³	A A A A J ³	J ² J ² J ² J ² J ⁴ J ² J ⁴ J ² J ³	J ² J ² J ⁴ J ² J ⁴ J ⁴ J ² J ² J ³	
FURANS	•			•	
2,3,7,8-TCDF 1,2,3,7,8-PeCDF 2,3,4,7,8-PeCDF 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 2,3,4,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	A A A A A A A J ³	A A A A A A A	J ¹ J ² J ² J ² J ⁶ J ²	J ¹ J ² J ⁶ J ²	

CIBA GEIGY CASE/SAS: 6684A-1 TABLE I CONT. RECOMMENDATIONS SUMMARY

SAMPLE NUMBER:	6684A-4/12907	6684A-4D/12908	6684A-5/12910	6684A-5/12912
DIOXINS				•
2,3,7,8-TCDD 1,2,3,7,8-PeCDD 1,2,3,4,7,8-HxCDD 1,2,3,6,7,8-HxCDD 1,2,3,7,8,9-HxCDD 1,2,3,4,6,7,8-HpCDD OCDD	A A A A A J ³	A A A A A J ³	A A A A A J ³	A A A A A J ³
FURANS 2,3,7,8-TCDF 1,2,3,7,8-PeCDF 2,3,4,7,8-PeCDF 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 2,3,4,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	A A A A A A A J ³	A A A A A A A	A A A A J ⁵ A A J ⁵ J ³	A A A A J ⁵ A A J ⁵ J ³

CIBA GEIGY CASE/SAS: 6684A-1 TABLE I CONT. RECOMMENDATIONS SUMMARY

SAMPLE NUMBER:	6684A-6/12914
DIOXINS	
2,3,7,8-TCDD 1,2,3,7,8-PeCDD 1,2,3,4,7,8-HxCDD 1,2,3,6,7,8-HxCDD 1,2,3,7,8,9-HxCDD 1,2,3,4,6,7,8-HpCDD OCDD	J ² J ² J ² J ² J ³
FURANS	
2,3,7,8-TCDF 1,2,3,7,8-PeCDF 2,3,4,7,8-PeCDF 1,2,3,4,7,8-HxCDF 1,2,3,6,7,8-HxCDF 2,3,4,6,7,8-HxCDF 1,2,3,7,8,9-HxCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,6,7,8-HpCDF 1,2,3,4,7,8,9-HpCDF OCDF	J ²

SUMMARY KEY

- A Accept the data
- J¹ Estimate (J) positive values and (UJ) non-detects for 2,3,7,8-TCDF for sample 6684A-3. 2,3,7,8-TCDF was not resolved from its 13C-2,3,7,8-TCDF analog.
- ${\tt J^2}$ Estimate (J) positive values and (UJ) non-detected values for all analytes associated with 6684A-3 and 6684A-6. The blank was contaminated during preparation.
- J³ Estimate (J) positive values for OCDD/OCDF due to low level contamination found in the blank for all samples.
- J⁴ Estimate (J) positive values and (UJ) non-detects for 1,2,3,6,7,8-HxCDF, 1,2,3,4,7,8-HxCDD, 1,2,3,6,7,8-HxCDD and 1,2,3,7,8,9-HxCDD for sample 6684A-3. The % RPD was outside the 50-150% range.
- J⁵ Estimate (J) positive values and (UJ) non-detects for 2,3,4,6,7,8-HxCDF and 1,2,3,4,7,8,9-HpCDF for sample 6684A-5. The % RPD was outside the 50-150% range.
- J⁶ Estimate (J) positive values for OCDF and 2,3,4,7,8-PeCDF due to % recoveries outside the control limits for the unspiked 6684A-3 samples.

DIOXIN DATA REVIEW WORKSHEET

	Ţ	PEM SAMPLES		
	Α.	Fortified Blank 083041 Compounds Found	Laboratory ID 12962 Concentration \$\rho \ell_{\rho}\$	EPA ID Laboratory No. 66844-1 G105125P02-01 Expected Concentration NA
	*'	1234678 HOCOD	100 140 58	N A
	B.	Fortified Std. WEELSC Compounds Found 2.3.7.8-TCDD	Laboratory ID 12901 Concentration 1.8	EPA ID Laboratory No. 66844-2 9145125P02-02 EPA 95% Prediction Level 1.8
	9	heponted value	within the	29% prediction limit.
	Tu l	beyond the 959: 2,3,7,8-TCDD in	the blank is reported by the blank is reported	the fortified standard is rel or a false positive for eported, reject all data. HrcPD/HrcDF, HPCPD/Hpc
but	. NO.	t at quantit	tation her	el which would
aff	ect	the avalys,	' 5 .	
*2	In	Therfering Hee	LDD/HXCDF	HPCDD/17xCDFand
		Do for on me	ne retenta	Which and
	ef	Lect the wa	tive analy	which did not
·			No Actio	

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Revision 0

II.	INITIAL AND	CONTINUING	CALIBRATIO	N .		
-	Date of 1	Initial Cal	ibration:	oct 9	1991	
	Date of (Continuing	Calibration	: oct	9 1991	oct 14, oct
<u>Date</u>	Instrument	\$ RSD. %D	Compound ()	Results)	Samples Af	fected 190
	· · · · · · · · · · · · · · · · · · ·					
						······································
		•				
						
Actio	on: % RSD >1 % D >30%	5% approxim approximat	ate all the ce all the a	associat ssociated	ed data (J data.	or UJ)
	Was a CC 12 hours	3 analyzed?	just prior t	co sample	analysis ar	nd every
		yes				
	•					
	A	11 crite	de e			
					•	•
				·		

Was a window defining marker solution analyzed with the case?

	First Isomer	Last Isomer
TCDD	23:62	29:62
PeCDD	33:39	38:92
HxCDD	42:63	46:20
HpCDD	50:77	52:15
TCDF	22:03	29:60
PeCDF	30:17	39:17
HxCDF	41.35	46:85
HpCDF	50:13	52:85

Were the PCDD/PCDF isomers reported within the defined windows? NO

Actions: If any of the PCDD/PCDF congeners were outside the retention window make sure that new descriptors are used.

AH MITERIANNI

6684A-3 SAMPLS

2,3,7,8-TCDF STAIL and U

Was a window defining marker solution analyzed with the case?

	First Isomer	Last Isomer
TCDD	23:55	29:52
PeCDD	33:36	38:87
HxCDD	42:68	46:15
HpCDD	<u> 50173</u>	_52113
TCDF	21:98	29:53
PeCDF	30:10	39:12
HxCDF	91:30	46:80
HpCDF	50:10	52:82

Were the PCDD/PCDF isomers reported within the defined windows?

60

Actions: If any of the PCDD/PCDF congeners were outside the retention window make sure that new descriptors are used.

All onitionia met.

Was a window defining marker solution analyzed with the case?

	First Isomer	Last Isomer
TCDD	36165	33:15
PeCDD	36165	41:17
HxCDD	45.25	48:67
HpCDD	53:08	54:43
TCDF	24155	33115
PeCDF	33175	42:02
HxCDF	44:03	49:32
HpCDF	52:50	55:15

Were the PCDD/PCDF isomers reported within the defined windows?

yes

Actions: If any of the PCDD/PCDF congeners were outside the retention window make sure that new descriptors are used.

411 contice met

T37	COLUMN	PERFORMANCE	RESOLUTION	CHECK
-----	--------	-------------	------------	-------

the CC3 period?	the chromatographic resolution of ¹³ C-Te solution for DB-5 columns calculated y <5 ¹³ C-2378-TCDD/ ¹³ C-1234-TCDD 5, 9, 0% between HxCDDs 32, 48, 49	QC limit ≤25%)
	331 columns: Not dowe . N A . 1478-TCDD/2378-TCDD	
_		(QC limit ≤25%)
	If the peak resolution is >25 %, the his/her professional judgement on the problem and its effect on the final r	e severity of the
۵.	Il criteria met.	

			Ŋ	=	R	1	IJ	Ab.	1
V.	METHOD	BLANKS	IJ	1	U	- 1		4	

Was a method blank prepared and analyzed for each matrix prior to analysis of samples?

Yes	[1]	No []	
Blank ID	Compound	<2% IS Signal	>2% IS Signal
·			•

Action: If a method blank associated with a sample group is contaminated, the associated positive samples and any sample containing any peaks that do not meet all identification criteria must be reextracted and reanalyzed.

1000 Recoveries of Fs - extanate 5 and US for SAMPCES 12903, 12904, 12905, 12906, SAMPCE Was corrophel.

v.	METHOD	BLANKS	ton	DF	B	112	•
----	--------	--------	-----	----	---	-----	---

Was a method blank prepared and analyzed for each matrix prior to analysis of samples?

Yes [$m{\mathcal{U}}_{j}$		ио []		
Blank ID	Compound OCPD	<2% IS Signal	>2% IS Signal	
		·		
				

Action: If a method blank associated with a sample group is contaminated, the associated positive samples and any sample containing any peaks that do not meet all identification criteria must be reextracted and reanalyzed.

All criteria met.

10W Level ocpologope

50 m Samples. 6684A-5! 12910, 6684A-6:12912

6684A-4:12907, and 6684A-4:12908

v. METHOD BLANKS OFBLK4

Was a method blank prepared and analyzed for each matrix prior to analysis of samples?

Yes [1,	ио[]	
Blank ID	Compound A 3 78 7cor	<2% IS Signal	>2% IS Signal
	12378 PecDF		
	133638 Hich 133638 Hich 133438 Hich		
	1237-84-A,CH		
	DOCAD DOCAD		
	HYCDE	•	
	OCDE		

Action: If a method blank associated with a sample group is contaminated, the associated positive samples and any sample containing any peaks that do not meet all identification criteria must be reextracted and reanalyzed.

BIK correspendenting sample prep. estimate 5+ and us for samples.

(6688A-6)

V. METHOD BLANKS

Was a method blank prepared and analyzed for each matrix prior to analysis of samples?

Yes	1.4	ио []	
Blank ID	Compound	<2% IS Signal	>2% IS Signal
•••••	-		
	-		

Action: If a method blank associated with a sample group is contaminated, the associated positive samples and any sample containing any peaks that do not meet all identification criteria must be reextracted and reanalyzed.

All chiteria mul

6684A-35/12906/12405

VI. MATRIX SPIKE

List percent recovery which did not meet the limits criteria.

Compound	MS & Recovery	Limits
TCDD	DOT DE	60-140
PeCDD	V 16H 165	60-140
HxCDD		60-140
HpCDD		60-140
OCDD		60-140
TCDF		60-140
PeCDF		60-140
HxCDF		60-140
HpCDF		60-140
OCDF	46 010 12705	60-140

Actions: Recheck calculations

No Action.

VI. MATRIX SPIKE 6684-35 12905

List percent recovery which did not meet the limits criteria.

	Compound	MS & Recovery	Limits
	Compound		60-140
	TCDD		60-140
	PeCDD		60-140
•	HxCDD		60-140
	HpCDD		60-140
	OCDD		60-140
	TCDF		60-140
	PeCDF		60-140
	HxCDF		60-140
	HpCDF		60-140
	OCDF	_ 46	60-140

Actions: Recheck calculations

no Action

jt and us Analyles.

VI. MATRIX SPIKE 4684A-35:12906

List percent recovery which did not meet the limits criteria.

Compound	MS & Recovery	Limits
TCDD		60-140
PeCDD		60-140
HxCDD		60-140
HpCDD		60-140
OCDD		60-140
TCDF		60-140
PeCDF	154	60-140
HxCDF		60-140
HpCDF		60-140
OCDF	4	60-140

Actions: Recheck calculations

NO ActiON

Page 31 of 36

VII. DUPLICATE

Was a duplicate run for each matrix?

Yes [1

No []

The RPD of each analyte detected must be within 50-150% range.

Recheck all calculations if beyond the specified Actions: range. Professional judgement should be used to

ascertain effect on final data.

6684A-4

OCDF 108

6684F5

6684A-3

123678 H, SDE -71

123478 HxCPD -200

123678 HXCDD -200

234678 144CDF -20

1234789 HPCDF-200

6684A-6 sample lost.

ext It for columns and in won detects.

VIII. RECOVERY STANDARD RESPONSE

•	CC-3 Standard ID:	Column:
Area Counts Upper Limit Lower Limit	¹³ C-1,2,3,4-TCDD	¹³ C-1.2.3.7.8.9-HxCDD
List samples which	did not meet criter:	ia.
	CC-3 Standard ID:	Column:
Area Counts Upper Limit Lower Limit	13C-1,2,3,4-TCDD	13C-1,2,3,7,8,9-HxCDD
List samples which	did not meet criter	ia.
	CC-3 Standard ID:	Column:
Area Counts Upper Limit Lower Limit	13C-1,2,3,4-TCDD	¹³ C-1,2,3,7,8,9-HxCDD
· · · · · · · · · · · · · · · · · · ·	did not meet criter	ia.
All crit-	eria mil.	

DIOXIN DATA REVIEW WORKSHEET

•	I III OAII DES		
A.	Fortified Blank	Laboratory ID	EPA ID Laboratory No.
	Compounds Found	Concentration	Expected Concentration
	Lost durin	ve extraction	v· ·
В.	Fortified Std. W #F 656	Laboratory ID	EPA ID Laboratory No.
•	Compounds Found	Concentration	[6684] 2 91US125702-01 EPA 95% Prediction Level
	2,3,7,8-TCDD	1, 4 14/9	1200/6
	1234678 HD CDD	3 6 NO 16	
	OCDE	0.15	

Action: If the 2,3,7,8-TCDD reported in the fortified standard is beyond the 95% prediction level or a false positive for 2,3,7,8-TCDD in the blank is reported, reject all data.

Interferences from Hycop/Hycop CHEROL, Hycop/Hycope

(NPTN) and OCDD/ & DF channel which.do not

after quantation of relive analytes

NO Action

x. Toxic equivalent factor (TEF) 6684A 2: 12901

Were samples with a TEF > 0.7ug/Kg for soil/sediment or fly ash; 7.0ug/Kg for chemical waste and 0.007ug/L for aqueous samples confirmed on SP-2300, SP-2331 or equivalent column?

Were EMPC values were included in the TEF calculations?

Yes [] No []

Check that the TEF values were calculated including EMPCs using the following guidelines:

Compound F	Multiplying actor	Conc		
2,3,7,8-TCDD	1.00	1800	1800	
Other TCDD	0.00			
2,3,7,8-PeCDD	0.50			
Other PeCDD	0.00			
2,3,7,8-HxCDDs	0.10			
Other HxCDD	0.00			
2,3,7,8-HpCDD	0.01	100	1.0	
Other HpCDD	0.00			
OCDD	0.001	3600	3. 6	
2,3,7,8-TCDF	0.100			
Other TCDF	0.000			
1,2,3,7,8-PeCI	F 0.050	· · · · · · · · · · · · · · · · · · ·		
2,3,4,7,8-PeCI	F 0.500			<u> موسوان بالقال فاساد</u>
Other PeCDF	0.000			
2,3,7,8-HxCDF	0.100			
Other HxCDF	0.000			
2,3,7,8-HpCDF	0.010			
Other HpCDF	0.000			
OCDF	0.001	150	~15	

Total Toxic Equivalent (800

Reference:

x. TOXIC EQUIVALENT FACTOR (TEF) 668419-1: 12902

Were samples with a TEF > 0.7ug/Kg for soil/sediment or fly ash; 7.0ug/Kg for chemical waste and 0.007ug/L for aqueous samples confirmed on SP-2300, SP-2331 or equivalent column?

Were EMPC values were included in the TEF calculations?

Yes [/]

No []

Check that the TEF values were calculated including EMPCs using the following guidelines:

	Compound	Multiplying Factor	Concentration Equivalen	Toxic t
	2,3,7,8-TCDD	1.00_		,
	Other TCDD	0.00		
	2,3,7,8-PeCDD	0.50		
•	Other PeCDD	0.00		
	2,3,7,8-HxCDD	s 0.10		
	Other HxCDD	0.00		
	2,3,7,8-HpCDD	0.01 10	1.0	
	Other HpCDD	0.00 19	0.0	
	OCDD	0.001 14	0 1,4	
	2,3,7,8-TCDF	0.100		
	Other TCDF	0.000		
	1,2,3,7,8-PeC	DF 0.050		
	2,3,4,7,8-PeC	DF 0.500		
	Other PeCDF	0.000		
	2,3,7,8-HxCDF	0.100		
	Other HxCDF	0.000		
	2,3,7,8-HpCDF	0.010		
	Other HpCDF	0.000 19	B 2700 0.0	
	OCDF	0.001 _5	8 0.058	

Total Toxic Equivalent 1.2

Reference:

x. TOXIC EQUIVALENT FACTOR (TEF) 66844-3: 12903

Were samples with a TEF > 0.7ug/Kg for soil/sediment or fly ash; 7.0ug/Kg for chemical waste and 0.007ug/L for aqueous samples confirmed on SP-2300, SP-2331 or equivalent column?

Were EMPC values were included in the TEF calculations?

Yes [] No []

Check that the TEF values were calculated including EMPCs using the following guidelines:

Compound	Multiplying Factor	Concentration Toxic Equivalent
2,3,7,8-TCD	D 1.00	
Other TCDD	0.00	
2,3,7,8-PeC	DD 0.50	1.0 4.5
Other PeCDD	0.00	
2,3,7,8-HxC	DDs 0.10	
Other HxCDD	0.00	
2,3,7,8-HpC		
Other HpCDD		D 0.63
OCDD	0.001 63	
2,3,7,8-TCD		140(R) 24
Other TCDF	0.000	
1,2,3,7,8 - P		1.9 0.24
2,3,4,7,8-P		16 23
Other PeCDF		
2,3,7,8-HxC		4, 7, 3, 2, 1 3, 8
Other HxCDF		
2,3,7,8-HpC		
Other HpCDF		
OCDF	0.001 3	0.039

Total Toxic Equivalent

53, 29

Reference:

6684A-3D/12904

X. TOXIC EQUIVALENT FACTOR (TEF)

Were samples with a TEF > 0.7ug/Kg for soil/sediment or fly ash; 7.0ug/Kg for chemical waste and 0.007ug/L for aqueous samples confirmed on SP-2300, SP-2331 or equivalent column?

Were EMPC values were included in the TEF calculations?

Yes [/] No []

Check that the TEF values were calculated including EMPCs using the following guidelines:

Compound Fa	Multiplying actor		ntration Toxic Equivalent
2,3,7,8-TCDD	1.00		,
Other TCDD	0.00		
1,2,3,7,8-PeCDD		. 9	
Other PeCDD	0.00		
2,3,7,8-HxCDDs		·	415
Other HxCDD	0.00		· · · · · · · · · · · · · · · · · · ·
2,3,7,8-HpCDD	0.01		
Other HpCDD	0.00		
OCDD	0.001 47	20	0,47
2,3,7,8-TCDF	0.100	2101	21
Other TCDF	0.000	`	······································
1,2,3,7,8-PeCD		4.7	0.24
2,3,4,7,8-PeCD		<u> 38 </u>	19
Other PeCDF	0.000		
2,3,7,8-HxCDF	0.100		
Other HxCDF	0.000		·
2,3,7,8-HpCDF	0.010		
Other HpCDF	0.000		
OCDF	ھُـ 0.001	12	0.022

Total Toxic Equivalent

494

Reference:

x. TOXIC EQUIVALENT FACTOR (TEF) 66844: 12907

Were samples with a TEF > 0.7ug/Kg for soil/sediment or fly ash; 7.0ug/Kg for chemical waste and 0.007ug/L for aqueous samples confirmed on SP-2300, SP-2331 or equivalent column?

Were EMPC values were included in the TEF calculations?

Yes [] No []

Check that the TEF values were calculated including EMPCs using the following guidelines:

Compound	Multiplying Factor	Concentration Toxic Equivalent
2,3,7,8-TCDD	1.00	
Other TCDD	0.00	
2,3,7,8-PeCI	D 0.50	
Other PeCDD	0.00	
2,3,7,8-HxCI	Ds 0.10	
Other HxCDD	0.00	
2,3,7,8-HpCI	D 0.01	
Other HpCDD	0.00	
OCDD	0.001_	84 0,084
2,3,7,8-TCDF		
Other TCDF	0.000	
1,2,3,7,8-Pe		
2,3,4,7,8-Pe		
Other PeCDF	0.000_	
2,3,7,8-HxCI		
Other HxCDF	0.000	
2,3,7,8-HpCI		·
Other HpCDF	0.000_	·
OCDF	0.001	

Total Toxic Equivalent

0.084

Reference:

X. TOXIC EQUIVALENT FACTOR (TEF)

6684A | 6684A-1

Were samples with a TEF > 0.7ug/Kg for soil/sediment or fly ash; 7.0ug/Kg for chemical waste and 0.007ug/L for aqueous samples confirmed on SP-2300, SP-2331 or equivalent column?

Were EMPC values were included in the TEF calculations?

Yes [] No [

Check that the TEF values were calculated including EMPCs using the following guidelines:

Compound	Multiplying Factor	Concentration Toxic Equivalent
2,3,7,8-TCD	D 1.00	
Other TCDD	0.00	
2,3,7,8-Pec	DD 0.50	
Other PeCDD	0.00	
2,3,7,8-HxC	DDS 0.10 3.4	- 35
Other HxCDD		
2,3,7,8-HpC	DD 0.01 3	5 0.036
Other HpCDD		
OCDD	0.001 8	0.081
2,3,7,8-TCD		
Other TCDF	0.000	
1,2,3,7,8-P	eCDF 0.050	
2,3,4,7,8-P		
Other PeCDF		
2,3,7,8-HxC		
Other HxCDF		
2,3,7,8-HpC		
Other HpCDF		A 199 199 199 199 199 199 199 199 199 19
OCDF	0.001 /	0 0.01

Total Toxic Equivalent

Reference:

"Interim Procedure for Estimating Risk Associated with Exposures to Mixtures of Chlorinated Dibenzo-p-dioxins and Dibenzofurans (CDDs and CDFs)" EPA/625/3-89/016.

0 44 0.13

6684H-5:12910

X. TOXIC EQUIVALENT FACTOR (TEF)

Were samples with a TEF > 0.7ug/Kg for soil/sediment or fly ash; 7.0ug/Kg for chemical waste and 0.007ug/L for aqueous samples confirmed on SP-2300, SP-2331 or equivalent column?

Were EMPC values were included in the TEF calculations?

Yes [] No [

Check that the TEF values were calculated including EMPCs using the following guidelines:

Compound	Multiplying Factor	Concentration Toxic Equivalent
 2.3.7.8-TCD	D 1.00	
Other TCDD	0.00	
2,3,7,8-PeC	DD 0.50	
Other PeCDD		
2,3,7,8-HxC	DDs 0.10	
Other HxCDD		
2,3,7,8-HpC	0.01 20	0.26
Other HpCDD	•	
OCDD	0.001 3	50 35
2,3,7,8-TCD	,	
Other TCDF	0.000	
1,2,3,7,8-F	eCDF 0.050	
2,3,4,7,8-F		
Other PeCDF		
2,3,7,8-HxC		
Other HxCDF		
2,3,7,8-HpC		
Other HpCDF		
OCDF	0.001	

Total Toxic Equivalent 0.6

Reference:

X. TOXIC EQUIVALENT FACTOR (TEF)

Were samples with a TEF > 0.7ug/Kg for soil/sediment or fly ash; 7.0ug/Kg for chemical waste and 0.007ug/L for aqueous samples confirmed on SP-2300, SP-2331 or equivalent column?

Were EMPC values were included in the TEF calculations?

Yes [\ No []

Check that the TEF values were calculated including EMPCs using the following guidelines:

Compound	Multiplying Factor	Concentration Toxic Equivalent
2,3,7,8-TCD	D 1.00	
Other TCDD	0.00	
2,3,7,8-PeC	DD 0.50	
Other PeCDD	0.00	
2,3,7,8-HxC	DDs 0.10	
Other HxCDD	0.00	
2,3,7,8-HpC	0.01 <u>2</u>	0.81
Other HpCDD	0.00	
OCDD	0.001_3	20 0.22
2,3,7,8-TCD		
Other TCDF	0.000	
1,2,3,7,8-P	eCDF 0.050	
2,3,4,7,8-P	eCDF 0.500	
Other PeCDF	0.000	-
2,3,7,8-HxC	DF 0.100	
Other HxCDF	0.000	
2,3,7,8-HpC	DF 0.010	
Other HpCDF	0.000	
OCDF	0.001	17 0.017

Total Toxic Equivalent のよる

Reference:

x.	TOXIC	EQUIVALENT	FACTOR	(TEF)	6684	A-	4	1291	4
							•		

Were samples with a TEF > 0.7ug/Kg for soil/sediment or fly ash; 7.0ug/Kg for chemical waste and 0.007ug/L for aqueous samples confirmed on SP-2300, SP-2331 or equivalent column?

Were EMPC values were included in the TEF calculations?

Yes [] No []

Check that the TEF values were calculated including EMPCs using the following guidelines:

Compound	Multiplying Factor	Concentration Equivalen	Toxic t
2,3,7,8-TCDD	1.00		
Other TCDD	0.00		· · · · · · · · · · · · · · · · · · ·
2,3,7,8-PeCDD	0.50		
Other PeCDD	0.00		
2,3,7,8-HxCDD	s 0.10		
Other HxCDD	0.00		
2,3,7,8-HpCDD	0.01 <u>4</u> み	6	40
Other HpCDD	0.00 200		
OCDD		MB 0-02	3
2,3,7,8-TCDF		<u> </u>	
Other TCDF	0.000		
1,2,3,7,8-PeC	DF 0.050 2	1	<i>H</i> 1 \ \
2,3,4,7,8-PeC	DF 0.500 <u>16</u>	7, 4	
Other PeCDF	0.000	•	
2,3,7,8-HxCDF	0.100 C	6.21 8.	7
Other HxCDF	0.000		
2,3,7,8-HpCDF	0.010 <u>16</u>	0,28 1.2	8
Other HpCDF	0.000		
OCDF	0.001	90 O.	190
•			

Total Toxic Equivalent

18-72/E 22 PE/L

Reference:

XI SAMPLE CALCULATION: FFF 0.87 TODD Q is = 2000 PZ

FONC 3,3,7,8-TCDD= 2000PE (214436570)/0.02079'Kg(14697787)0.87 x 106pg

 $\frac{\text{FIDE}}{\text{EDL}} = 3.5 \times 200092 \left[(0.20 - 0.05) \right] 9.5 \times 10^{3} + (.19 - 0.06) \left[0^{4} \right] \left[0.43 \right] = 0.43 \text{ pps}$ = 1.61 m//s = 0.43 pps = 0.43 pps = 0.43 pps

EMPC = 1,2,3,4,6,7,8-HpcDF = (1.05 ×108 + 1.0 ×108) x2100 Pg = 2700 pg/g

Concentration (ng/g) = $\frac{\text{Ois } \times (Ax^1+Ax^2)}{(\text{W or V}) \times (Ais^1+ais^2) \times RRFX \times D}$

EDL = $\frac{2.5 \times \text{Ois } \times (\text{Hx}^1 + \text{Hx}^2) \times D}{(\text{W or V}) \times (\text{His}^1 + \text{His}^2) \times \text{RRFx}}$

EMPC = Ois x (Ax^1+Ax^2) x D W or V x (Ais^1+Ais^2) x RRFx

where:

Qis = quantity (ng) of appropriate internal standard added to sample before extraction

Ax¹ and Ax² =integrated areas of the two quantitation ions
W and V = weight (g) or volume (L) of sample extracted
RRFx = calculated relative response factor from the continuing calibration

 Hx^1+Hx^2 = peak heights of the noise for the quantitation ions His^1+His^2 = peak heights of the internal standard quantitation ions

D = dilution

Lockheed Engineering & Sciences Company

M/3-9202 J Originator

198 Croeby Drive Bedford, MA 01730

PHONE CONVERSATION RECORD

Conversation with:	Date 2 15 192
Name Jill Sphencen	Time / / / C AM PM
Company Vijstuac	
Address Company	Originator Placed Cal Originator Received Cal
Phone <u>5:3-1-24-277</u> 3	W.O. NO
Subject	
Notes: Jeff sprensor - rep	wested DC-2 forms
- he told me all offer	n internation wood
be sont out today	
	D C
Resport T Faxed out	UC-2 toms to
Dels spronser	
Sponce VIII Requished In	former flow NUS receive
b. Fol X av Fok	5 1992
File	Follow-Up-Action:
Follow-Up By: Copy/Route To:	
	Originator's Initials



B-92-01-77
Originator

198 Crosby Drive Bedford, MA 01730

PHONE CONVERSATION RECORD

Conversation with:	Date / /28/9)
Name Seff Sprenger	Time /2:10 AM/PM
Company 1ce y steme	
Address Onego N	Originator Placed CallOriginator Received Call
Phone 503 - 624 - 2773	W.O. NO
Subject	
Notes: <u>To GG. Spronsen</u> retur	red my call she
he soid he would try t	o set all the
required Into tome o	in mardey the
1992	
	·
	· · · · · · · · · · · · · · · · · · ·
VFile	Follow-Up-Action:
Follow-Up By: Copy/Route To:	:
	Originator's Initials